



Memorandum

To: Dante Rodriguez, Remedial Project Manager
Site Cleanup Section 3, SFD-7-3
USEPA Region 9

Through: Joe Eidelberg, Chemist
Quality Assurance Section, EMD-3-2
USEPA Region 9

From: Kathy O'Brien, Project Manager *Keo*
ICF, Environmental Services Assistance Team (ESAT) Region 9

ESAT Contract No.: EP-W-13-029
Technical Direction No.: 10106165

Date: July 7, 2017

Re: Review of Analytical Data, **Tier 3**

Attached are comments resulting from ESAT Region 9 review of the following analytical data:

Site:	Anaconda Yerington Copper
Site Account No.:	09 GU QB08
Case No.:	46764
SDG No.:	MYA9K7
Laboratory:	Chemtex Environmental Laboratory (CHX)
Analysis:	CLP Metals by ICP-AES and ICP-MS
Samples:	6 Soil Samples
Collection Dates:	February 15 and March 13 and 15, 2017
Reviewer:	Anna Pajarillo, ESAT

EXES Data Manager has been updated with the results of this review and the validation level revised to S3VEM; the dynamic deliverables were regenerated and are available on the SMO Portal.

If there are any questions, please contact Joe Eidelberg (QA Program/EPA) at (415) 972-3809.

Attachment

cc: Richard Freitas, CLP COR USEPA Region 9
Raymond Flores, CLP COR USEPA Region 6

CLP PO: ☒ FYI ☐ Action

SAMPLING ISSUES: ☒ Yes ☐ No

10106165-20049/46764/MYA9K7 Rpt

Data Validation Report – Tier 3

Case No.: 46764
SDG No.: MYA9K7
Site: Anaconda Yerington Copper
Laboratory: Chemtex Environmental Laboratory (CHX)
Analysis: CLP Metals by ICP-AES and ICP-MS
Reviewer: Anna Pajarillo, ESAT
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I. SDG SUMMARY

For Sample Information and Laboratory Quality Control (QC), refer to EXES National Functional Guidelines (NFG) data validation reports *Analytical Sample Listing* and *Inorganic Analytical Sequence*. EXES Data Manager has been updated with the results of this review and the validation level revised to S3VEM; the dynamic deliverables were regenerated and are available on the SMO Portal. The data qualifier definitions, as described in page 6 of the National Functional Guidelines, are attached to this report.

Field QC

Field Blanks (FB): None.
Equipment Blanks (EB): None.
Background Samples (BG): None.
Field Duplicates (D1): MYA9K7 and MYA9H7 (in SDG MYA9H7).
Field Duplicates (D2): MYA9K8 and MYA9J7 (in SDG MYA9H7).

CLP PO Action

None.

Sampling Issues

1. Samples were collected on February 15 and March 13 and 15, 2017 and were shipped to the laboratory on March 20, 2017 (see Additional Comments).
2. The SDG Narrative indicates that the samples were received in a box and a temperature blank was not present.
3. The sample log-in sheets indicate that custody seals were not present on the box.

Additional Comments

The samples were analyzed for Contract Laboratory Program (CLP) metals. Aluminum, calcium, iron, manganese, potassium, and sodium were analyzed by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES). Antimony, arsenic, barium, beryllium, boron, cadmium, chromium, cobalt, copper, lead, magnesium, molybdenum, nickel, selenium, silver, thallium, thorium, uranium, vanadium, and zinc were analyzed by Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS) under Modification Analysis 2725.0.

The chain of custody record (COC) indicates that the samples were collected on February 15 and March 13 and 15, 2017 and were shipped to the laboratory on March 20, 2017. The reviewer presumed that the samples were stored properly from the time of collection to the time of shipping. The laboratory documentation indicates that they were received at a temperature of six degrees Celsius.

For the ICP-MS analysis, true values and recoveries for molybdenum were incorrectly reported on Form 4 (ICP Interference Check Sample). The laboratory submitted revised Form 4 upon request, on June 30, 2017. Recoveries for molybdenum are within the QC limit.

On Form 5A for the ICP-MS analysis, arsenic (74%) did not meet the 75-125% QC criterion for matrix spike sample recovery and was not reported on the post digestion spike sample recovery Form 5B. The laboratory submitted revised Form 5A upon request, on June 30, 2017. The recovery for arsenic (75%) is within the QC limit and post digestion was not required.

All standards and spiking solutions were analyzed before the expiration date.

This report was prepared in accordance with the following documents:

- ☐ USEPA Contract Laboratory Program Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration), ISM02.4, October 2016;
- ☐ ICP-MS Analysis Plus Boron, Molybdenum, Thorium, and Uranium, Modified Analysis 2725.0, February 8, 2017; and
- ☐ USEPA National Functional Guidelines for Inorganic Superfund Data Review, January 2017.

For technical definitions, refer to *Exhibit G (Glossary of Terms)*, *USEPA Contract Laboratory Program Statement of Work for Inorganic Superfund Methods (Multi-Media, Multi-Concentration)*, ISM02.4.

II. VALIDATION SUMMARY

The data were evaluated based on the following parameters:

	<u>Parameter</u>	<u>Acceptable</u>	<u>Comment</u>
1.	Data Completeness	Yes	
2.	Preservation and Holding Times	Yes	
3.	ICP-MS Tune Analysis	Yes	
4.	Calibration	Yes	
	a. Initial	Yes	
	b. Initial and Continuing Calibration Verification	Yes	
5.	Laboratory Blanks	Yes	B
6.	Field/Equipment Blanks	N/A	
7.	ICP Interference Check Sample (ICS)	Yes	
8.	Laboratory Control Sample (LCS)	Yes	
9.	Duplicate Sample Analysis	Yes	
10.	Spike Sample Analysis	No	C
11.	ICP Serial Dilution	Yes	
12.	ICP-MS Internal Standards	Yes	
13.	Analyte Quantitation	Yes	A, E
14.	Field Duplicate Sample Analysis	No	D
15.	Overall Assessment of Data	Yes	

N/A = Not Applicable.

III. VALIDITY AND COMMENTS

- A. Results above the method detection limit (MDL) but below the contract required quantitation limit (CRQL) are estimated and flagged "J." Results are considered qualitatively acceptable but quantitatively unreliable due to uncertainties in analytical precision near the quantitation limit.
- B. The following results are qualified as non-detected (U) due to low level initial calibration blank (ICB) and continuing calibration blank (CCB) contamination.
 - ☐ Boron in samples MYA9K8 and MYA9P5.
 - ☐ Selenium in sample MYA9P5.
 - ☐ Silver in sample MYA9K7.

Analyte amounts greater than the MDL but less than the CRQL were reported in calibration blanks and preparation blank at the concentrations presented below.

Analyte	Blank	Concentration, µg/L
Boron	ICB/CCBS43	4.1/1.2
Selenium	ICB/CCBS44	0.70/0.85
Silver	ICB/CCBS44	0.22/0.20

Sample results that are greater than or equal to the MDL but less than or equal to the CRQL are reported as non-detected (U) at the respective CRQL.

- C. The following results are estimated and flagged “J-” or “UJ” because matrix spike recoveries are outside the method QC limit.

☐ Antimony and thorium in all field samples.

Matrix spike recoveries for the analytes listed above in QC sample MYA9K7S do not meet the 75-125% criterion for accuracy as presented below.

Analyte	% Recovery
Antimony	35
Thorium	73

The detected results for analytes listed above are considered quantitatively uncertain and may be biased low.

The following post-digestion spike recoveries were reported in QC sample MYA9K7A. The post-digestion spike recoveries do not reflect the entire sample preparation and analysis; the impact on reported results cannot be determined. Qualification is based on the matrix spike recovery only.

Analyte	Post-Digestion Spike, % Recovery
Antimony	121
Thorium	68

Since both the post- and pre-digestion spikes do not meet the QC criteria for thorium, the unacceptable pre-digestion spike recovery may indicate poor laboratory technique or matrix effects which may interfere with the analysis.

- D. Results for the following field duplicate pair do not meet the relative percent difference (RPD) criterion for precision as presented below.

	MYA9J7	MYA9K8		
Analyte	(D1), mg/kg	(D1), mg/kg	RPD	QC Limit
Nickel	3.1	3.9	23%	20%

This uncertainty should be evaluated in the context of project data quality objectives to determine data usability.

- E. Sample MYA9K7 was reanalyzed at a 2.0-fold dilution due to manganese concentration exceeding the calibration range. The result for manganese in this sample is reported from the diluted analysis.

DATA QUALIFIER DEFINITIONS FOR INORGANIC DATA REVIEW

The definitions of the following qualifiers are prepared according to the document, "National Functional Guidelines for Inorganic Superfund Data Review," January 2017 (Table 1, page 6).

- U The analyte was analyzed for, but was not detected above the level of the reported sample quantitation limit.
- J The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample.
- J+ The result is an estimated quantity, but the result may be biased high.
- J- The result is an estimated quantity, but the result may be biased low.
- UJ The analyte was analyzed for, but was not detected. The reported quantitation limit is approximate and may be inaccurate or imprecise.
- R The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample.